doi:10.1017/S1431927616005651
Microsc. Microanal. 22 (Suppl 3), 2016

Ultra High Energy Resolution EELS Mapping using Aberration-corrected Low-voltage STEM Equipped with Monochromator

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We have been developing a low-voltage analytical electron microscope working at an accelerating voltage of from 60 kV to 15 kV, which enables us to observe and analyze carbon-related materials with less sample damage by irradiation of electrons. This microscope, which employs a double Wien-filter monochromator system [1], is equipped with delta-type aberration correctors for probe- and image-forming lens system [2]. The double Wien-filter monochromator, which is located between the extraction anode of Schottky source and the accelerator, enables us to obtain an achromatic monochromatic probe. Therefore the energy spread of electrons after the monochromation is controllable by choosing the width of the slit which is located between two filters, independently on the probe size at the specimen. The delta-type aberration corrector consists of triple dodeca-poles to correct the fifth-order aberration including six-fold astigmatism as well as the spherical aberration. In addition, so as to detect the high energy resolution spectra in electron energy-loss spectroscopy (EELS) by the monochromated electron source, the microscope is equipped with a high energy resolution spectrometer (Quantum-ERS from Gatan Inc.), which incorporates with the highly sensitive detection system at lower accelerating voltage and the highly stabilized power supplies for the prism and the lens system.

Figure 1 (a) ~ (c) show raw ADF-STEM images of single-layered graphene at 60 kV and their Fourier transforms obtained with different settings of the monochromator. These three images were obtained at 60 kV (a) by a non-monochromatic probe with energy spread of 700 meV, by a monochromatic probe with (b) 228 meV with a 4 μm-width slit and (c) 134 meV with a 2 μm-width slit. As shown in these STEM images obtained at a low-voltage, the monochromation of electron probe improves spatial resolution due to a small chromatic aberration. By using a highly monochromatic 60-kV electron probe with energy spread of 134 meV, C-C dumbbells of single-layered graphene were clearly resolved. The Fourier transforms of two images in (b) and (c), which were obtained with monochromatic electron probes of 228 meV and 134 meV, show an isotropic resolution limits. Thus, the double Wien-filter type monochromator enables us to obtain a round monochromated electron probe at any energy resolution due to achromatic focus.

We tested the EELS map in the low-loss region on a hexagonal boron nitride (h-BN) specimen obtained with a monochromatic probe with a 0.1 μm-width slit at an accelerating voltage of 30 kV. The experimental conditions were a 1 nm probe size, a 10 pA probe current and a 0.3 seconds acquisition in each pixel. Figure 2 (a) shows the ADF-STEM image from the EELS mapping area. Fig. 2 (b) shows the extracted low-loss spectrum from the yellow square area at the edge of the specimen shown in Fig. 2 (a). This spectrum, which was measured with an energy resolution of 22 meV, showed a sharp peak corresponding to an optical phonon at about 170 meV. Figure 2 (c) shows the EELS map at the phonon excitation energy, whose intensity is normalized with the zero-loss peak intensity in each pixel. This EELS map suggests that the phonon intensity is distributed in not only the specimen area but the vacuum area beyond the edge of the specimen with the distance of more than 100 nm, due to the delocalization of inelastic scattering.

References

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[3] This work is supported by JST, Research Acceleration Program (2012–2016).

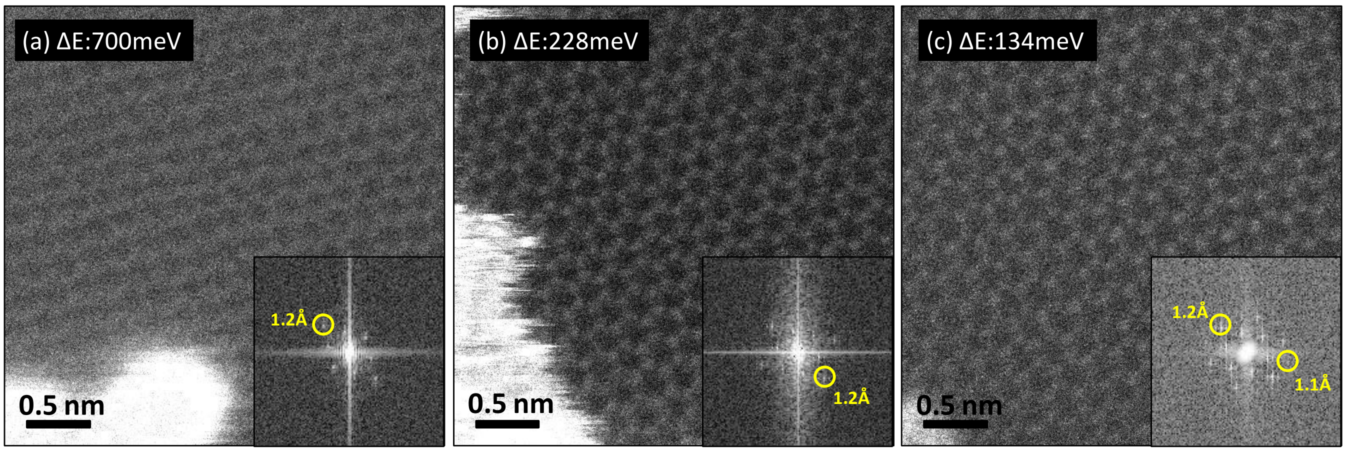

Figure 1. Raw ADF-STEM images from single-layer graphene and their power spectra obtained at 60 kV (a) by an non-monochromatic probe with energy spread of 700 meV, by a monochromatic probe with (b) 228 meV by using 4 μm slit and (c) 134 meV using 2 μm slit. These three images in (a) ~ (c) were obtained with the convergent semi-angle of 33 mrad and the probe current of 28 pA, 10 pA and 5 pA, respectively.

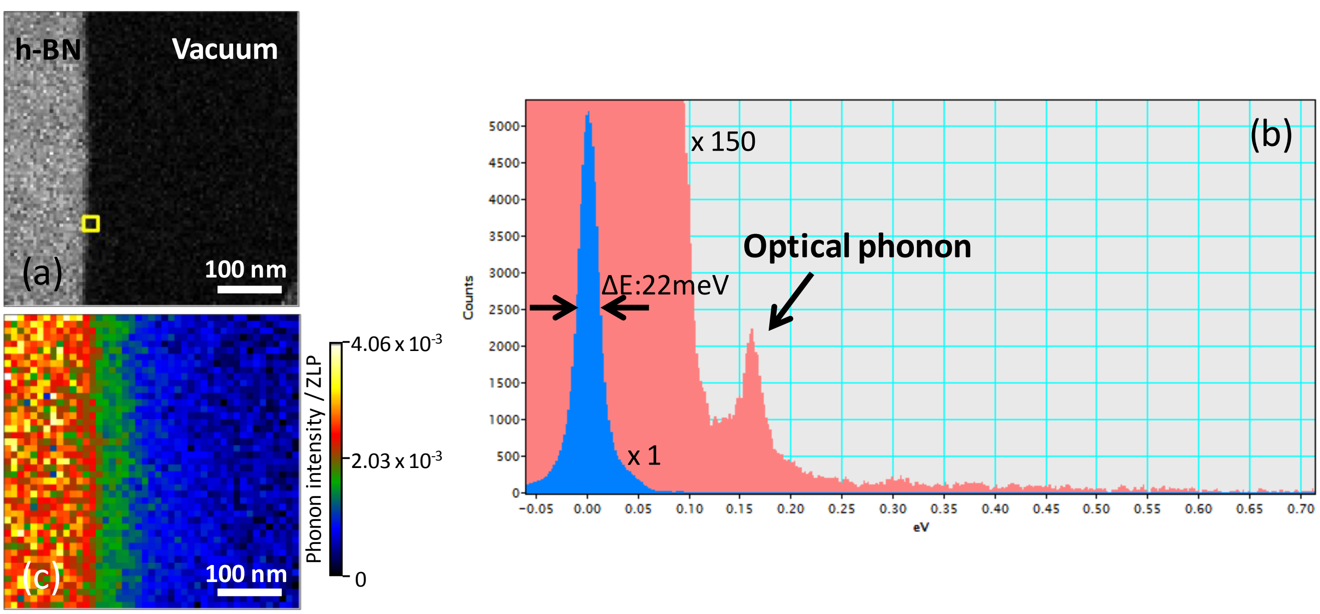

Figure 2. (a) ADF-STEM image of h-BN from the mapping area, (b) extracted low-loss spectrum obtained from the yellow square area at the edge of the specimen in Fig. 2(a) and (c) EELS phonon map, whose intensity is normalized with the zero-loss peak intensity in each pixel.